FABRICATION OF POLYMER DERIVED SILICA BONDED ALUMINA CERAMICS

A Thesis Submitted

In partial fulfillment of the requirement

For the degree of

BACHELOR OF TECHNOLOGY

Submitted by:

BHUKYA SUDHAKAR

ROLL NO-110CR0581

SUPERVISOR:

PROF. SHANTANU KUMAR BEHERA

DEPARTMENT OF CERAMIC ENGINEERING,

NATIONAL INSTITUTE OF TECHNOLOGY ROURKELA, ROURKELA-769008

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CERTIFICATE

This is certified that the work contained in the project entitled **"FABRICATION OF POLYMER DERIVED SILICA BONDED ALUMINA CERAMICS"** by Bhukya Sudhakar (Roll 110CR0581) in partial fulfilment of the requirements of the award of Bachelor of Technology Degree in Ceramic Engineering at the National Institute of Technology, Rourkela is an authentic work carried out by him under my supervision and guidance.

To the best of my knowledge, the matter embodied in the thesis has not been submitted to any other university / institute for the award of any Degree or Diploma

SHANTANU KUMAR BEHERA
PROFESSOR
Department of Ceramic Engineering
National Institute of Technology
Rourkela-769008

Date:

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I also thank to all the faculty members of ceramic engineering department for their suggestions during this project work. And finally, my hearty thanks to all my friends who have constantly helped me.

Bhukya Sudhakar

110CR0581

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ABSTRACT:

Present work focuses on the polymer derived silica bonded alumina ceramics. Sintering has been done at 1500^{o}C and 1600^{o}C. The alpha-alumina phase of (305 nm from ALMATIS&449 nm of TRL alumina) micron particle size has been used as starting material. Along with Polymethylsiloxane or puresilica is added with isopropylalcohol. The study of the densification of pellet at varying soaking time of 1, 2, 4, and 6 hours has also been carried. For better densification mixing is done under magnetic stirring. The microstructure of the sintered body is analyzed and densification due to a grain growth has been observed. We are successful to minimize the porosity up to 0.7% and preparing a dense product. The Vickers hardness test has been carried out for testing of mechanical property and was found to have good hardness value.

CONTENTS

CHAPTER 1

INTRODUCTION

PDCs - Polymer Derived Ceramics

1. Introduction

Polymer Derived Ceramics (PDCs), as the name suggests, represents a synthesis route for the realization of ceramic components through the controlled pyrolysis of polymeric precursors. Silicon based polymeric precursors demonstrated to be excellent candidates for the realization of many technologically important ceramic components such as fibers, coatings, in- filtrated porous media and complex bulk parts. After these discoveries and first successful experiments, the growing interest for this new methodology led to further significant improvements of their chemistry, synthesis, processing and properties. Many different classes of pre ceramic polymers have been synthesized in the last decades, the most important being polysiloxanes, polysilazanes and polycarbosilanes. With this methodology it was possible to obtain not only binary ceramics such as Si_3N_4 or SiC, but also more complex compositions in the SiOC and SiCN systems. Increasing the sophistication of the starting precursors.

The relatively low cost of the precursors, the wide variety of compositions achievable, the characteristic microstructure (which is, in most cases, impossible to be achieved by conventional methods), the unique and exceptional thermo-mechanical and chemical properties of the final ceramics, the possibility of shaping the precursors using well-established, conventional polymer forming technologies such as Polymer Infiltration Pyrolysis (PIP), injection molding, coating, extrusion, Resin Transfer Molding (RTM), fiber drawing and many others, makes this methodology an extremely promising route for a relatively simple realization of ceramic components.

Applications of PDCs: Because of their physical-chemical and functional properties as well as their ability of being shaped using a wide variety of processing methods, PDCs have

found application in several key fields such as information technology, transport, defense, energy as well as environmental systems, biomedical components and micro or nano electromechanical systems.

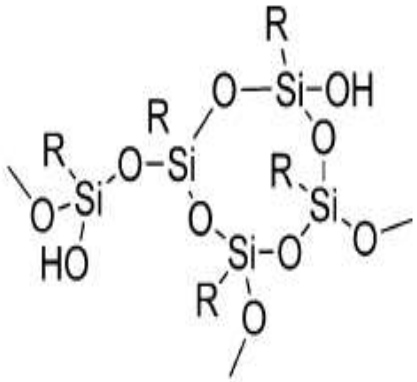
POLYMETHYL SILOXANE:

Polysiloxanes are surely the most important class of preceramic polymers. The main reasons for their widespread utilization are related to their generally low cost (the lowest among all Si-based polymers), their easy and cheap synthesis route, and finally their interesting thermo-chemical stability, which makes this class of precursors very versatile, easy to handle and processable under normal conditions without any particular precaution.

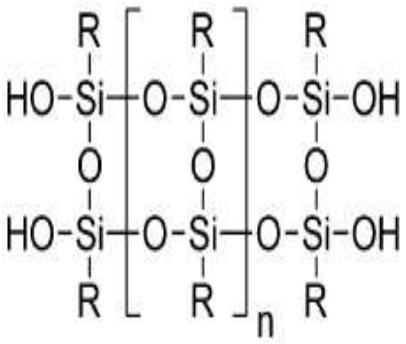
Besides their use as preceramic precursors, they are widely available in other more common applications like sealants, lubricants, adhesives and gaskets. They are generally order- less, colorless, water resistant, chemical resistant, electrically insulating and stable at high temperatures. Their higher thermal stability, together with relatively high melting and boil-ing points, make this class of polymers the preferred choice when organic polymers are not applicable. Typical applications are as sealants, coolants in transformers, long lasting motor-insulation, lubricants for bearings, foam-control agent in laundry detergents or as coatings to protect facades and historical monuments.

Polysiloxanes are widely used even in other high-tech fields like aerospace industry, or as protecting materials in the semiconductor industry, or during the processing of products like optical glass fibers, silicon wafers and chips. They are widely used as adhesion promoters in glues, sealants, pigments and paints, but also in the textiles and rubber industry. Moreover, their extremely low reactivity generally makes them non-toxic, and for this reason they can be used also in the personal care products industry, in biomedical applications like breast implants, or even as oral anti-foaming agent (e.g. simethicone) or as food additives.

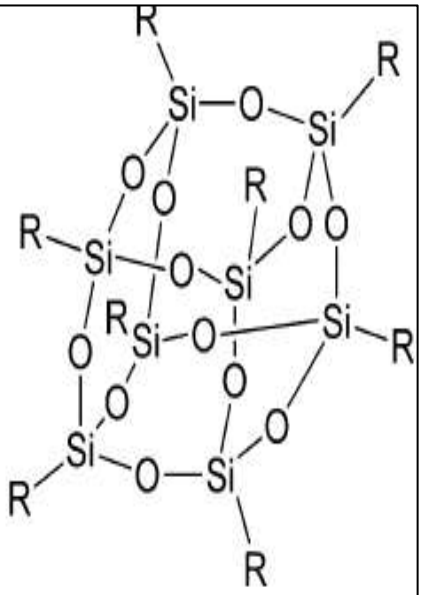
Their extremely interesting properties and the continuous development of this class of polymers, polysiloxanes market is expected to further grow and to find new applications, like for example as high oxygen permeable contact lenses, adhesive foams, synthetic fabric, waterproof membranes, process aids, or in lithographic applications.



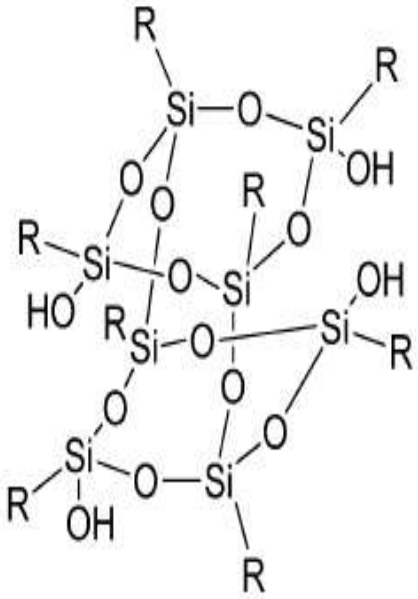
Random structure



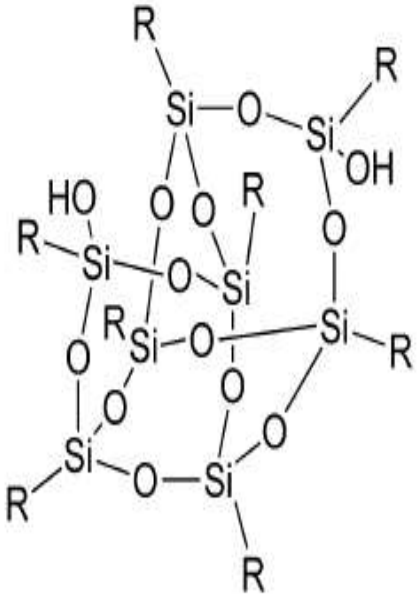
Ladder structure

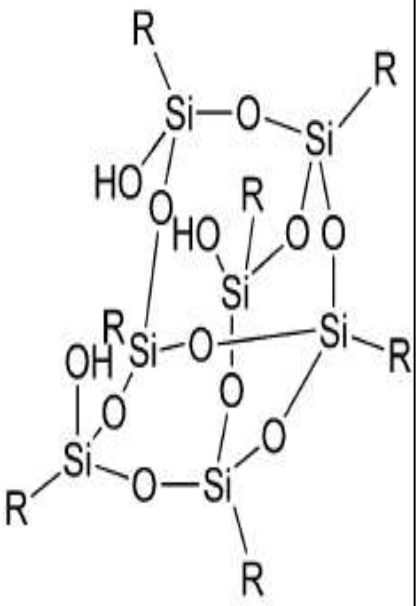


Cage structure



Double-decker structure





Partial cage structures

ALUMINA:

Alumina is a versatile material used as refractory, engineering ceramics material, abrasive and in various other applications where chemical inertness coupled with its high hardness and abrasiveness. Alumina may also be called aloxide, aloxite, or alundum depending on particular forms or applications. In recent years ceramics have attracted great scientific and technological attention due to an improvement in mechanical properties such as hardness, strength and wear resistance, along with the possibility of super plasticity, as compared with the monolithic matrix material .It may be useful as structural and functional materials for a variety of applications in different fields of technology.

MULLITE:

In the last years Mullite with the composition$3Al_2O_3.2SiO_2$ in particular and the Al_2O_3–SiO_2 system in general have become challenging materials in the fields of structural and optoelectronic applications. The special features of Mullite based ceramics are related to its high refractoriness and its good thermo mechanical properties, low thermal expansion and conductivity, as well as to its excellent oxidation and heat resistance.

Mullite is an excellent candidate for advanced ceramic applications such as gas filters, heat exchangers, multilayer packaging, and window material in the mid-infrared range. It is also used in ceramic fibers and matrices of ceramic matrix composites e.g., used for the fabrication of furnace burners, catalytic converter substrates, and for thermal protection systems for combustors in gas turbine engines.

Mullite is rarely found in nature because of its high temperature and low pressure formation conditions. The occurrence of Mullite is a result of post Caledonian volcanic activities in which high temperature Mullite phases are deposited when clay minerals are heated through contact with magma. Mullite belongs to aluminum silicate family with an orthorhombic structure. The Mullite crystal structure consists of a three-dimensional framework of alternate corner sharing of the AlO6 octahedral and SiO4 (or AlO4) tetrahedral. The aluminum silicate crystal structure can be modified into various orthorhombic structures ranging from Sillimanite $Al2O_3.SiO_2$ to $4Al_2O_3 \cdot SiO_2$, achieved by substituting Si4+ ions with Al3+ ions in the tetrahedral sites of the alternating aluminum and silicon columns and the introduction of ordered oxygen vacancies.

CHAPTER 2

LITERATURE REVIEW

Alumina (Al_2O_3) has wide application as engineering ceramic material due to its high hardness value. It has high melting point (2054 °C), low thermal expansion and high compressive strength which provides good thermal shock resistance [5]. Alumina has good electrical insulation at high temperatures, good wear resistance and high hardness, makes it suitable for components such as ball valves, piston pumps and deep drawing tools. For machining and grind of alumina diamond tools are needed. Alumina can be used in pure form and also in alloying component in aluminum oxide based ceramics which contain greater than 85% Al_2O_3. Commonly, the green density effects the sintering of the product. The green density can be controlled with pressure in powder pressing. Some properties of Al_2O_3 are listed in Table 2.1. [5]

Properties	**Unit**	**Alumina**
Density	g/cc	3.98
Tensile strength	Mpa	300-900
Hardness	Hv	2200
Young modulus	GPa	380
Fracture toughness	$MPa.m^{1/2}$	4.40

Table 2.1 Mechanical properties of Al_2O_3 [5]

Grain size distribution is one of the most crucial parameters in the successful sintering of alumina. Packing characteristic is another critical parameter for sintering. The particle shapes can be needlelike, spherical, tabular, platelet or uniaxial. The best packing of alumina powders is obtained using spherical shapes of various sizes randomly distributed in the batch.

G. L. Messing et al [6], has reported that Mullite in a diphasic mixture consisting of a pre ceramic polymer filled with alumina nanoparticles was investigated and The key findings are the addition of nano sized fillers to pre ceramic polymers allows to obtain Mullite ceramics at a low temperature, with very favorable kinetics and a high degree of microstructural control on the

crystalline phase assemblage. Fabrication methods can be used with this precursor mixture, and with considerable processing advantages for the realization of shaped components.

Ralf Riedel et al [7] , A new strategy has been followed to synthesize Mullite Sic based ceramics using alumina or functionalized alumina nano sized particles as filler together with a available polymer. Mullite formation was achieved at unusual low temperatures due to the high reactivity of the nano alumina filler particles towards the silica rich ceramic matrix formed from the polysiloxane fraction. This novel process enables the formation of dense Mullite Sic nano composites with relatively low porosity using polymer forming techniques. The microstructural results indicate that Sic as well as the segregated carbon hinders the growth of the Mullite crystals.

Helen Reveron et al [8], Has observed that The addition of small quantities of colloidal silica to a commercial alumina powder has a significant effect on its densification and microstructure evolution. At higher temperatures, silica allowed the formation of an intergranular liquid phase which leads to abnormal grain growth in the final stage of sintering (1550–1650 °C). And an increase of sintering shrinkage rates was observed (nomaly called by us "P") follows the formation of the liquid phase. Nevertheless, densification to the theoretical density was then prevented because some pores remain trapped within or between the abnormally grown large grains of a particle.

Hotta et al [9] studied that grain size and relative density of the sintered compacts and It was observed that microstructure of sintered compacts without acid washing was found to have discontinuous grain growth. The heterogeneous grains became bigger with rise of sintering temperature. On the other hand, the grains of the sintered compacts with acid was found homogeneous, even at higher sintering temperature also Then the relative densities of the sintered compacts with acid wash were higher than that of the sintered compacts without acid-wash. With increasing sintering temperature, and the difference of the relative densities of the sintered compacts. Thus, ceramic compacts with a fine grained and uniform microstructure is desirable for ceramic applications to produce a reliable structural part that it can be easily obtained by acid treatment of the slip-casted body.

Paolo Colombo *et a l*[10]reported that Porosity can be engineered in polymer-derived ceramics

by employing several processing methods, which include replica of a polymeric template, and direct foaming of a solution or slurry or the use of sacrificial pore formers. Components with hierarchical porosity can also be produced either by controlled pyrolysis, and deposition of various meso porous layers etching or the addition of suitable fillers. The produced ceramic components, possessing various compositions, morphologies and properties, have been tested with success in several diverse applications.

Hans Joachim Klee be et al[11])discussed the fundamental understanding of the relationship between the architecture of the pre ceramic precursor and the composition and microstructure of the resulting PDC-NCs is needed. In Systems have to be developed and systematically investigated with respect to cross linking and ceramization processes with their microstructure evolution upon polymer to ceramic transformation.

Enrico Bernardo et al[12] has proposed approach for the production of luminescent materials from pre ceramic polymers containing nano sized oxide particles is certainly attractive, and because of the easy shaped of the pre ceramic polymer matrix could be exploited to produce luminescent coatings, or luminescent structured (e.g. porous) bodies and 12in analogy to silicones without fillers.

Rodrigo Moreno et al[13] has observed that The effect of the dispersants on the rheological behavior of concentrated suspensions was also studied and The sintered densities and the microstructures of specimens obtained by slip casting were studied, demonstrating that Mullite powders obtained by a single step combustion process can be processed through conventional shaping routes.

Brian Derby et al [14] has studied that Mullite is believed to form by an inter diffusion reaction between Al2O3 and SiO2 containing material. The size of the Si inclusions that result from this redox process scale with the microstructure, which is in turn controlled by the Al particle size.

OBJECTIVE OF THE WORK

Aim of the present work is to develop dense ceramic specimen from using different alumina sources and silica sources (eg. Silres MK polymer & pure silica). This work focuses on enhancement of density as well as mechanical property of ceramic and particle size with increasing temperature. It also discusses how a small (2-10 vol %) amount of silica source affects the final density and mechanical properties of polymer derived Silica bonded with alumina.

CHAPTER 3

EXPERIMENTAL WORK

3. EXPERIMENTAL WORK

Raw alumina powder was procured from TRL & ALMATIS Pvt. Ltd.

3.1 XRD ANALYSIS

The presence of alumina phase in the used powder is known from the X-ray diffraction measurement of powder. When X-rays passes through matter, the radiation interacts with the electrons in the atoms, resulting in scattering of the radiation as shown in Figure 3.1. For crystalline materials the distances between the planes is same and if the atoms are of the same magnitude as the wavelength of the X-rays, constructive and destructive interference will occur. This diffraction where X-rays are emitted at characteristic angles based on the spaces between the atoms organized in crystalline structures are called planes.

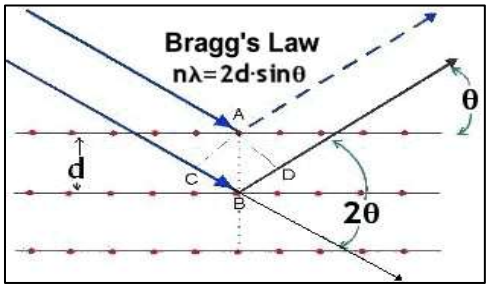


Figure 3.1: Study of crystallographic plane during XRD

3.2 PARTICLE SIZE DISTRIBUTION

A suspension of alumina powder is prepared with 0.3 gram alumina powder in 50ml distilled water. This suspension is kept for ultrasonication for 15 minutes. The temperature of the SUSPENSION IS maintained below 35°C. Ultrasonic energy is given to the suspension for homogeneous separation of suspended particles. This suspension is injected into a cuvette and particle size is analyzed. The plots of particle size distribution versus intensity and volume is observed.

Principle:

Laser diffraction measures particle size distributions by measuring the angular variation in intensity of light scattered as a laser beam passes through a dispersed particulate sample. Large particles scatter light at small angles relative to the laser beam and small particles scatter light at

large angles. The angular scattering intensity data is then analyzed to calculate the size of the particles responsible for creating the scattering pattern, using the Mie theory of light scattering.

3.3 BATCH PREPARATION:

BATCH-1: 90%ALUMINA (TRL) and 10%MK (POLYMETHYLSILOXANE)

For this batch of 10cc solution of IPA in which alumina is 18.905 gm of 9.5cc and silica from poly methyl siloxane (MK)is 0.665gm of 0.5cc solution.in this poly methyl siloxane gives 75% silica.

BATCH-2: 90% ALUMINA (almatis) and 10%MK (POLYMETHYSILOXANE)

For this batch of 10cc solution in which alumina is 17.91 gm of 9.0cc and silica from poly methyl siloxane (MK) is 1.333 of 0.5cc solution.in this poly methyl siloxane gives 75% silica.

BATCH-3: 95% ALUMINA (TRL) and 5%MK (POLYMETHYLSILOXANE)

For this batch of 10cc solution in which alumina is 17.91 gm of 9.0cc and silica from poly methyl siloxane (MK) is 1.333 of 0.5cc solution.in this poly methyl siloxane gives 75% silica.

BATCH-4: 95%ALUMINA (ALMATIS) and 5%MK (POLYMETHYlSILOXANE)

For this batch of 10cc solution in which alumina is 18.905 gm of 9.5cc and silica from poly methyl siloxane (MK) is 0.665gm of 0.5cc solution.in this poly methyl siloxane gives 75% silica

BATCH-5: 95%ALUMINA (ALMATIS) and 5%PURE SILICA

For this batch of 10cc solution in which alumina is 18.905 gm of 9.5cc and pure silica is 0.575gm of 0.5cc solution.

BATCH-6: 98%ALUMINA (ALMATIS) and 2%MK (POLY METHYL SILOXANE)

For this batch of 10cc solution in which alumina is 19.502 gm of 9.8cc and pure silica is 0.23gm of 0.2cc solution. in this poly methyl siloxane gives 75% silica

BATCH-7: 98%ALUMINA (ALMATIS) and 2%PURE SILICA

For this batch of 10cc solution in which alumina is 19.502 gm of 9.8cc and pure silica is 0.267gm of 0.2cc solution.

3.4 Mixing and Grinding:

Pre ceramic polymer consist of a poly methyl siloxane in powder form and alumina is also a powder. The polymer was dissolved in acetone under magnetic stirring for 15 minutes, thus producing a solution.aftter that alumina powder were added under magnetic stirring to the

solution by volume percent. The mixture was producing a stable and homogeneous dispersion of alumina nano particles, in which no sedimentation was observed. The dispersion was poured in to a glass container and dried at 60 ^{O}C overnight. After evaporation of solvent a solid silicone alumina nano composite mixture was obtained, in which nano sized filler was homogeneously distributed. After that mixture was finely grinded up to fine powder obtained.

3.5 Cross-linking:

The cross-linking of the pre ceramic polymer is of fundamental importance for the obtainment of a material that must result unmeltable during the pyrolysis step at higher temperature, when the polymer converts into the final ceramic material. After that powder cross link in air at 400 ^{O}C alumina and polymer of silica hold tightly together.

3.6 Milling and Pressing:

For Cross linked powder to fine powder milling is done for 10 minutes at 150 rpm speed. Then powder samples of weight(0.6 to 0.7)gm was prepared then pellet pressing is done using poly vinyl alcohol(PVA) as binder and for cleaning mold acetone was used. For pressing operation at 3.5 ton of load and dwell time of 60 seconds.

3.7 Drying and Sintering:

Pellets are dried under atmospheric condition for a period of around 24hrs at RT. Removal of liquid occurs at this stage. Then the pellets are kept in oven at 110^{o}C for complete removal of liquid (~99%) for a period of about 24hrs.After this sintering of green pellets are done at under different temperatures. The sintering has been done according to the following sintering profile shown in Figure 3.7. The images of sintered pellets are show in Figure 3.8 below.

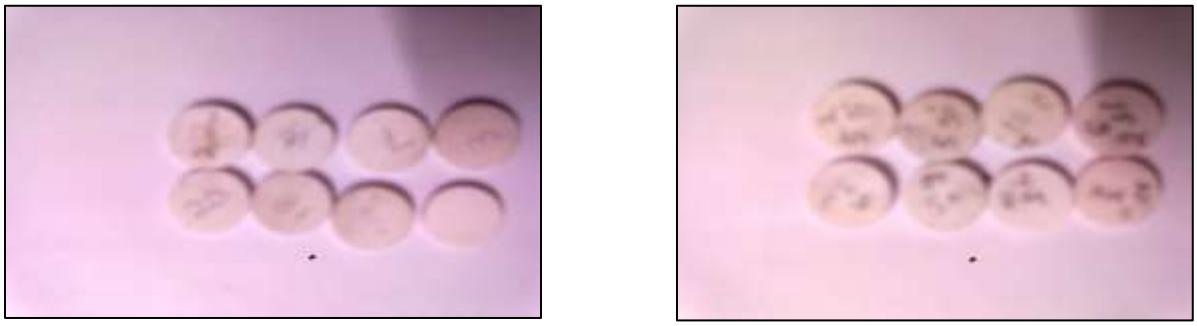


Figure: 3.2 Sintered Pellets at different sintering durations

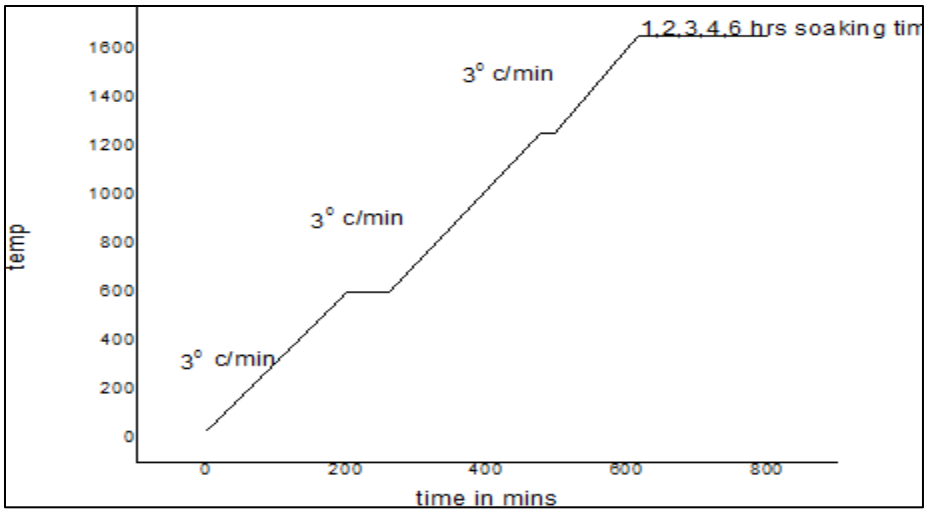


Figure 3.3: Temperature profile during sintering process

Flowchart of experimental work:

MK (or pure silica) Is added to 10ml IPA(isopropanol) under magnetic stirring for 15 minutes

⇩

Alumina (TRL OR ALMATIS) is added to the above solution

⇩

Drying over night at 60 ^{O}C

⇩

Grinding is done to get a fine powder mixture

⇩

Crosslinking at 400 ^{O}C

⇩

Milling is done for 10 minutes at 150 rpm speed

⇩

Weighing (0.6-0.7) gm and pressing at3.5 ton of load and dwell time of 60 seconds

⇩

Drying & sintering at1500, 1600 for (1hr,2hr,3hr,4hr,6hr)soaking time

⇩

Characterizations

3.8 CHARACTERISATION OF PHYSICAL PROPERTIES:

As known, inspection and testing of refractory is important to ensure the use of the end product. Apparent porosity (AP), bulk density (BD), relative density and percent volume shrinkage is determined to check the physical properties. Furthermore, mechanical testing (Brazilian disc test, Hardness and fracture toughness) and microstructure analysis is carried

3.8.1 DETERMINATION OF AP,BD, &RD:

BD determines the overall weight coming upon the foundations i.e. strength. A good specimen should have low porosity & high bulk density. The green sample fired at 1500^{o}C, 1600^{o}C (pellets) are weighed with 0.01 gm accuracy. This is called dry weight (D). The dry specimen is placed in a beaker and is filled with kerosene. This beaker is kept in desiccator for 2 hours & evacuated to a pressure of less than 25 mm of Hg. After that samples are taken out. Test specimen is suspended with the help of pan in kerosene and suspended weight of sample in water is taken which is called suspended weight (S). Now liquid drops appearing on the surface of sample are wiped & weight is taken in air. This is called soaked weight (W).

Calculations:

The apparent porosity (AP) and bulk density (BD) is calculated as:-

$$\mathbf{AP} = \frac{W-D}{W-S} \times 100\ (\%)$$

$$\mathbf{BD} = \frac{D}{W-S} \times \textit{density of the media in which expt. is performed}$$

Here, kerosene is used as media (density of kerosene is = 0.81 g/cc).

RD = (density of substance/true density)*100

3.9 MICROSTRUCTURAL ANALYSIS:

Firstly the samples are cut into small pieces; these pieces are rubbed on diamond grinding cloth in order to make its surface plane. These samples are mounted with the help of Bakelite followed by hot pressing at temperature around 135^{o}C for 3 minutes and load is applied of 20 Kilo Newton (KN). Then three consecutive polishing is done to the sample (6µm, 3 µm, and 1 µm cloth) using diamond suspension spray. Ultra cleaning of samples is performed after each

polishing. For this purpose samples are immersed into the solution of the distilled water and soap oil. Mounted sample is put on a heater for burn out of Bakelite. Thermal etching is done for each sample in order to get separated grain boundaries so that better microstructure is obtained

3.10 EVALUATION OF MECHANICAL PROPERTY:

3.10.1 Vicker's hardness:

Hardness valued were determined by Vicker's indentation technique. For each sample, at least 3 impressions were produced, and the average value was taken as the final hardness value.

Figure 3.5: Vickers Hardness testing machine

Vickers Hardness is a very popular test, which is characterized by a square based diamond pyramid indenter, exactly ground to a standard form with 136 degrees between opposite faces and used to leave a mark in metal under a precisely applied force by taking care to avoid impact: the diagonals of the impression have to be measured using a suitable microscope and the results are either calculated using a given formula for each of the forces (loads) used.

The formula used for calculating the Vickers Hardness Number (or Diamond Pyramid Hardness)

$$HV_P = 1.8544 * P/ d2$$

Where, P = force (load) in kilograms

d = diagonal length of the impression in mm (millimeters) (or, better, average of two readings, mutually perpendicular).

CHAPTER 4

RESULTS AND DISCUSSION

4.1 XRD ANALYSIS:

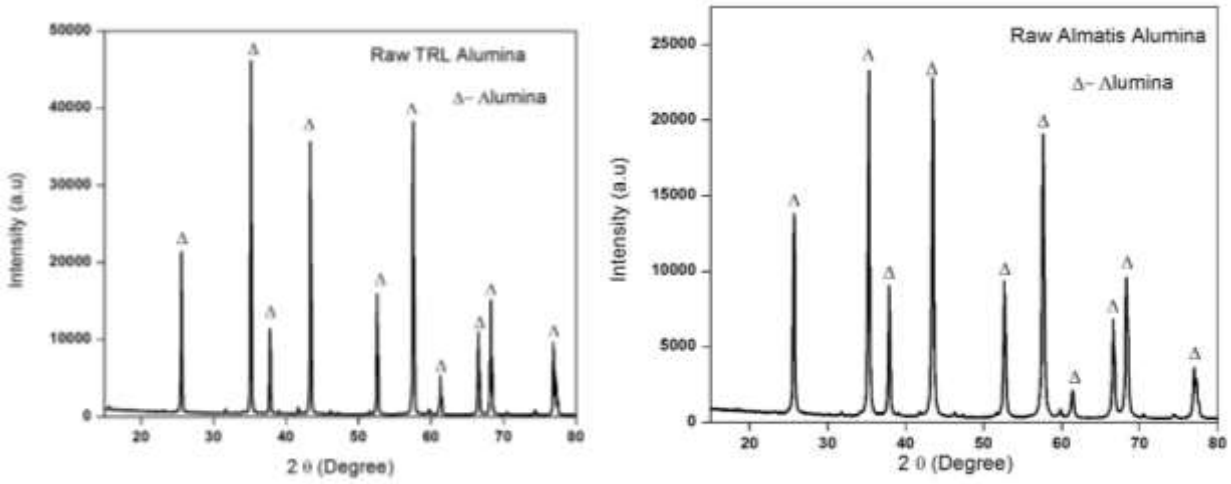


Fig.4.1: XRD pattern of Raw alumina powders

Fig.1 shows the XRD pattern of Alumina powders obtained from TRL Krosaki ltd. and Almatis A16 Grade respectively. The peaks were labelled and Phase pure Alumina is found as confirmed from Ref code: 82-1467.

4.2 PARTICLE SIZE DISTRIBUTION:

Particle size distribution helps us to understand about the average particle size and range of the particles present. Fig. 4.2(A) shows the particle size distribution of the raw alumina powder. The particle size measurement reveals mono-modal size distribution. Average particle size has been found to be 1.107 micron and the particle size has been found to be in the range of 0.825 micron to 1.718 micron from the figure.

It can be observed that the maximum volume particles (~30.5%) are finer than 1 micron (Table 4.1). The statistical graph for particle size distribution also shows the similar results as shown in Fig. 4.2(B)

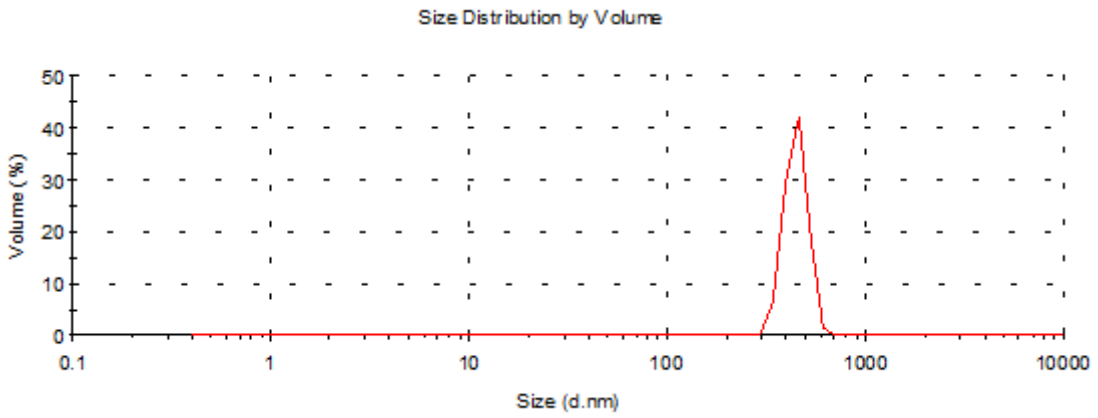


Fig. 4.2 PSD of Crosslinked TRL Alumina Powder TRL

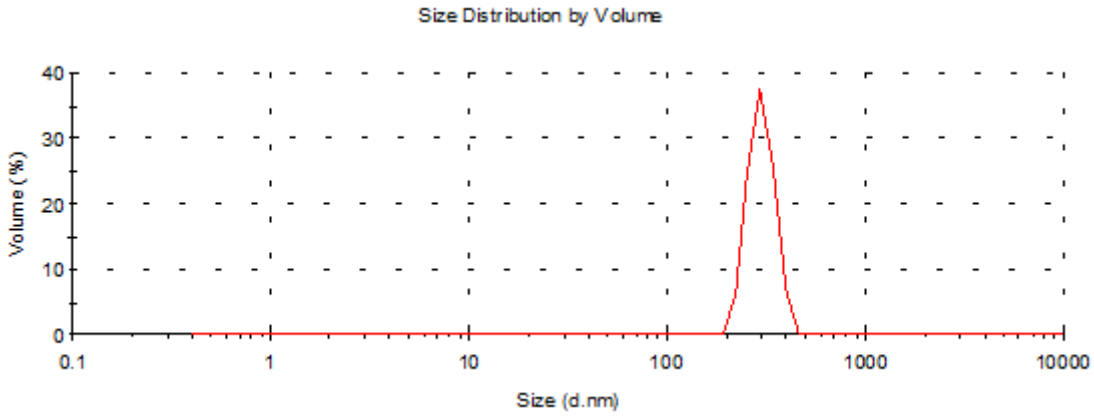


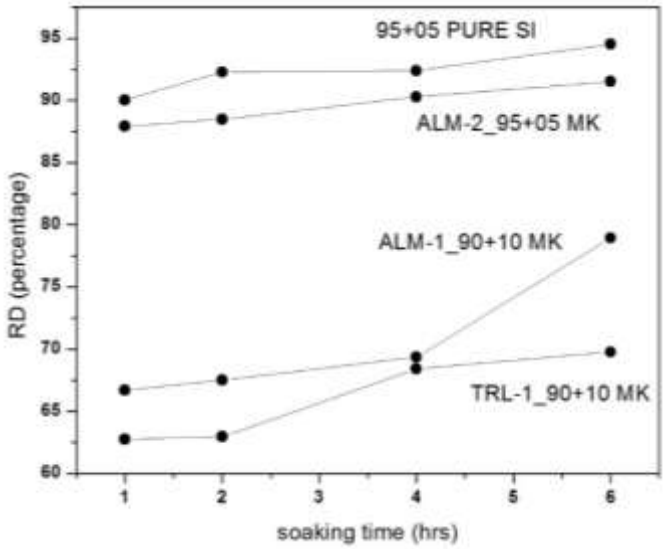
Fig.4.2.3 PSD of Crosslinked Almatis Alumina Powder

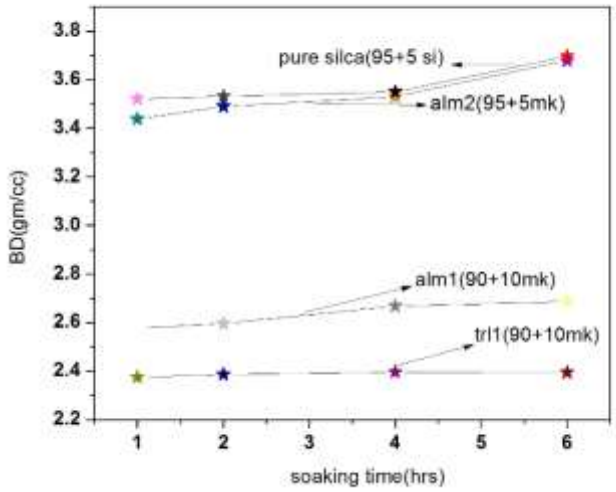
4.3 APPARENT POROSITY, BULK DENSITYAND RELATIVE DENSITY of SINTERED SPECIMENS:

4.3.1 Compaction and Sintering of cross linked powders:

Taking the optimized composition of powder, the green pellet specimens are sintered at 1500^{o}C with 1, 2, 4 and 6 hours to see the variation in density of alumina specimen with respect to soaking time. The soaked samples (alumina(trl)-90&mk-10,alumina(almatis)-90&mk-10, alumina(almatis)-95&mk-5, alumina(almatis)-90&pure silica-10) at 2, 4 ,6 and 10 hours soaking

is respectively. Table 4.3 shows the AP and BD of the sintered specimens. It was observed that as the sintering time is increased the density also raises and the apparent porosity minimizes. As the soaking time increases the complete sintering of specimen occurs. We can see that the maximum density (3.6964g/cc) and minimum porosity (5.655%) is for 6 hour soaking and the minimum density (2.3753) & maximum porosity (39.25%) is for 2 hour soaking as represented in Fig. 4.5.





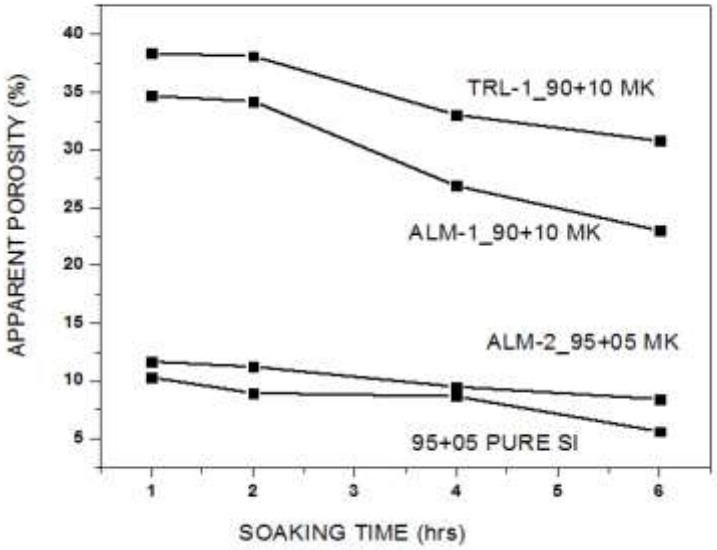


Figure 4.3.1 Dependence of Bulk Density and apparent porosity to soaking time for sintered samples at1500^{O}Cfor 1hr2hr,4hr,6hr

4.3.2 Sintered at 1600^{o}C:

The green pellet specimens are sintered at 1600^{o}C with 2, 4 and 6 hours to see the variation in density of alumina specimen with respect to soaking time. The soaked samples alumina(trl)-90&mk-10,alumina(almatis)-90&mk-10, alumina(almatis)-95&mk-5, alumina(almatis)-90&pure silica-10) at 2, 4 and 6 hours soaking is respectively. Table 4.3.2 shows the AP and BD of the sintered specimens. It was observed that as the sintering time is increased the density also raises and the apparent porosity minimizes and relative density increasing as well. As the soaking time increases the complete sintering of specimen occurs. We can see that the maximum density (3.8324g/cc) and minimum porosity (0.67%)and relative density(96.96%) is for 6 hour soaking and the minimum density (3.64& maximum porosity (3.40%) and minimum relative density (93.31%)is for 2 hour soaking as represented in Fig. 4.3.2

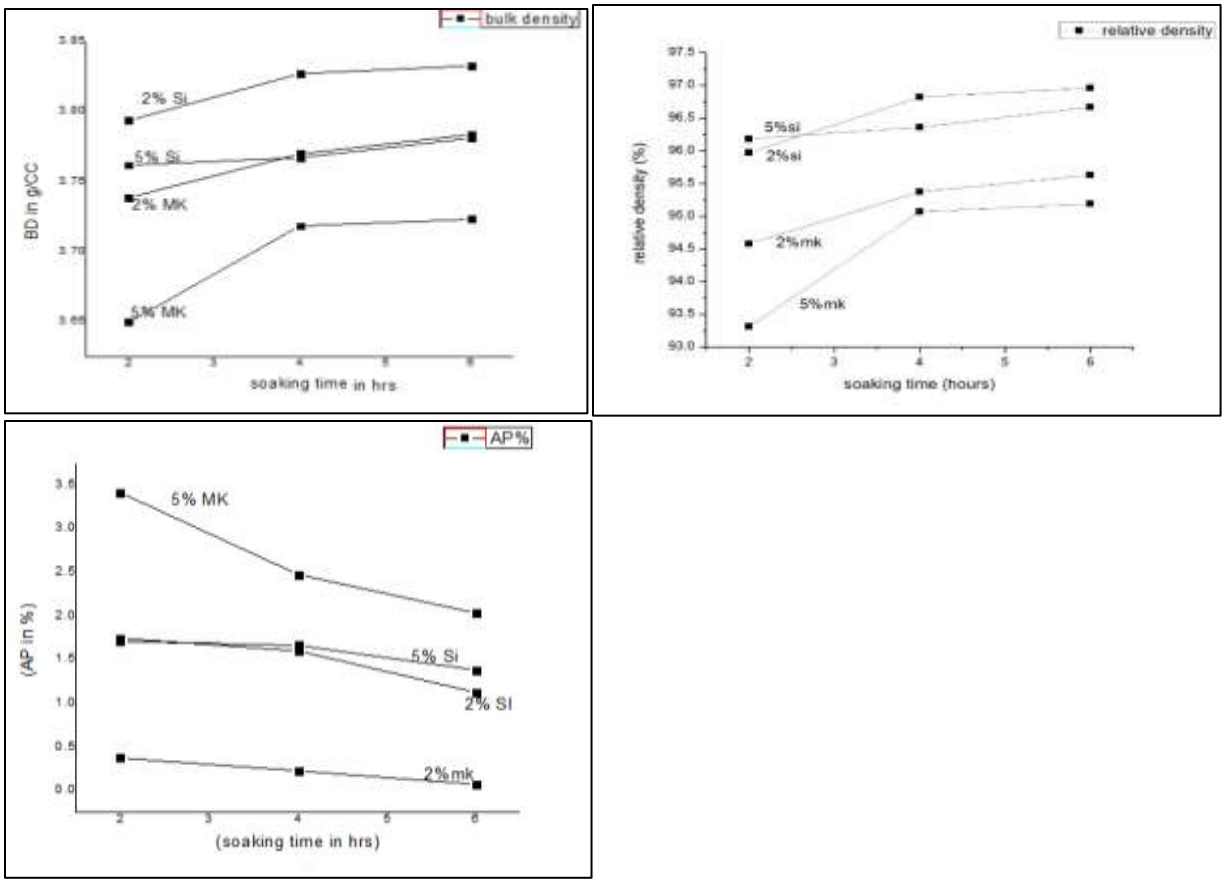


Figure 4.3.2 Dependence of Bulk Density and apparent porosity to soaking time for sintered samples at1600^{O}Cfor2hr,4hr,6h**r**

4.4 MICROSTRUCTURE STUDY:

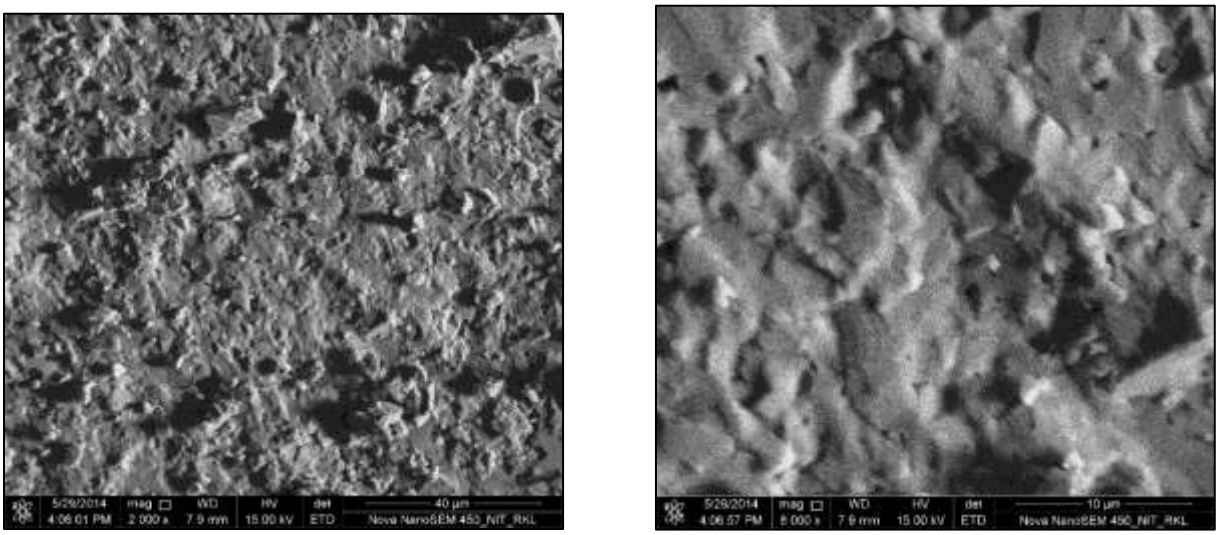


Fig:2 MK6(98%alumina& 2%mk)

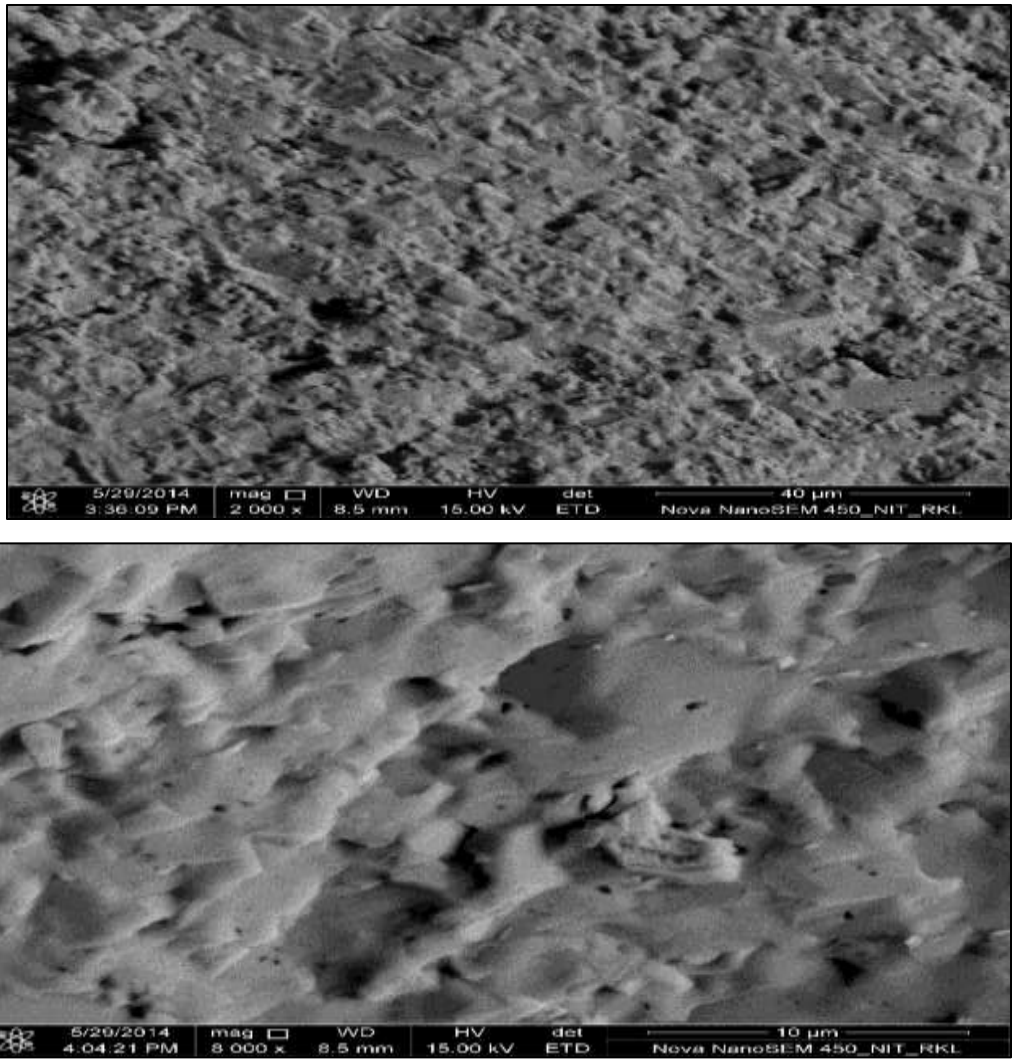


Fig:5 MK6 (95%alumina& 5%Mk)

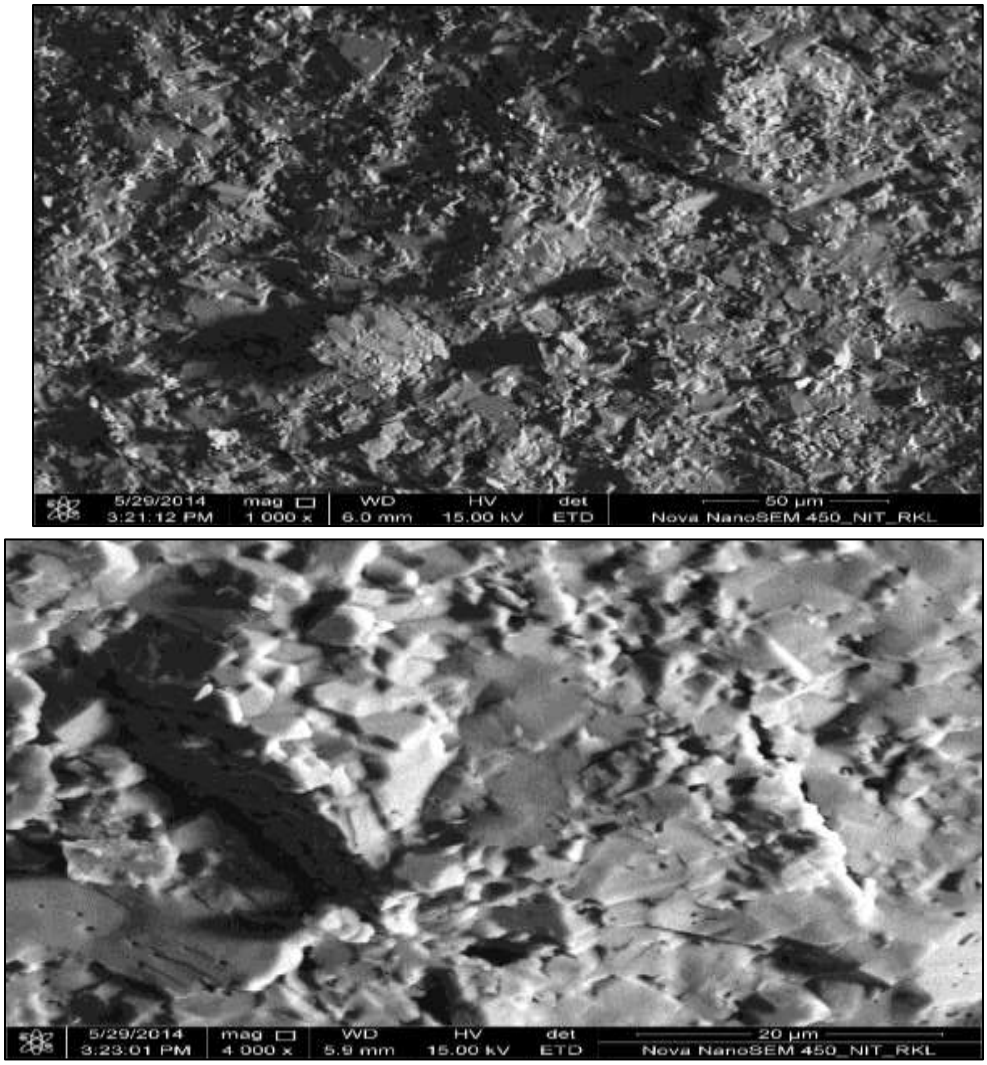


Fig: 2 Si6 (98%alumina& 2%pure silica)

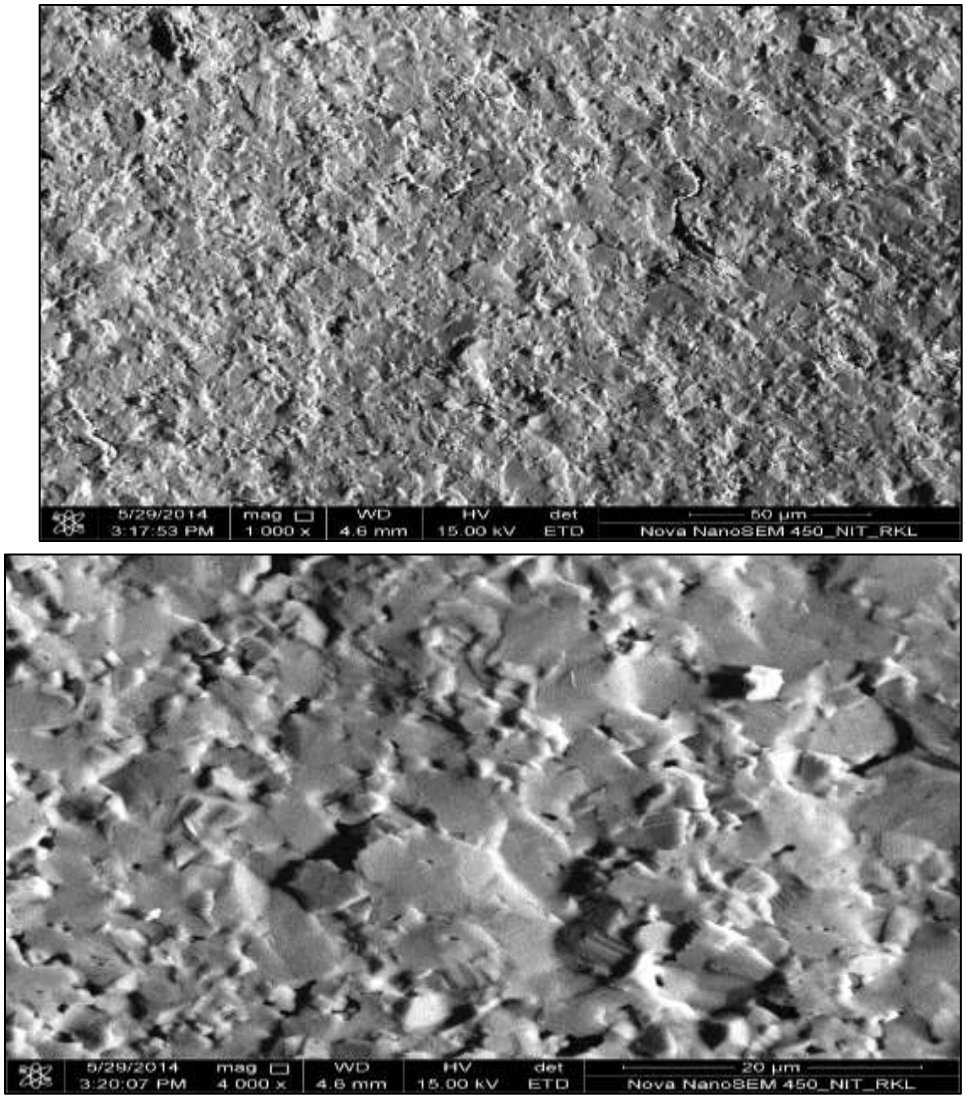


Fig: 5 Si6 (95%alumina& 5%pure silica)

Fig 4.6: FESEM micrographs of the sintered samples at 1600^{O}C for 2MK, 5MK,2SI and 5SI for 6 hours

4.5 Dilatometer Analysis: For high amount of silica content produces lower shrinkage and low amount of silica produces higher shrinkage

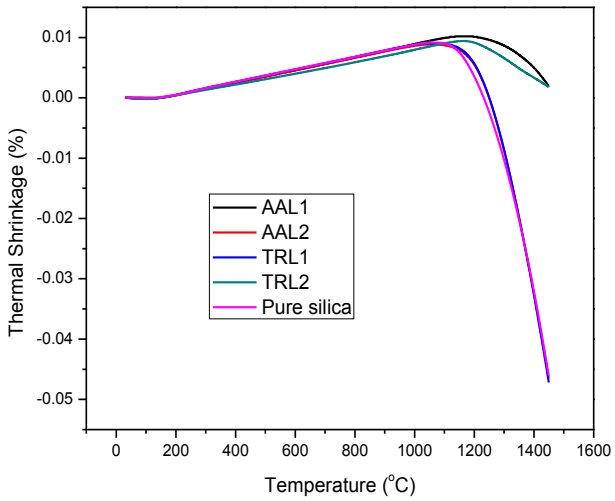


Fig4.5: Dilatometer Analysis

SAMPLE NAME	COMPOSITION	SHRINKAGE (%)	TEMPARATURE(^{O}C)
BATCH-1	90-Al_2O_3,10-MK	1	1171
BATCH-2	90-Al_2O_3,10-MK	1	1200
BATCH-3	95 Al_2O_3,5-MK	4	1118
BATCH-4	95- Al_2O_3,5-MK	4	1118
BATCH-5	95 Al_2O_3,5-SI	4	1118

Table: 4.5 shrinkage values from dilatometer analysis

4.6 Vickers hard ness:

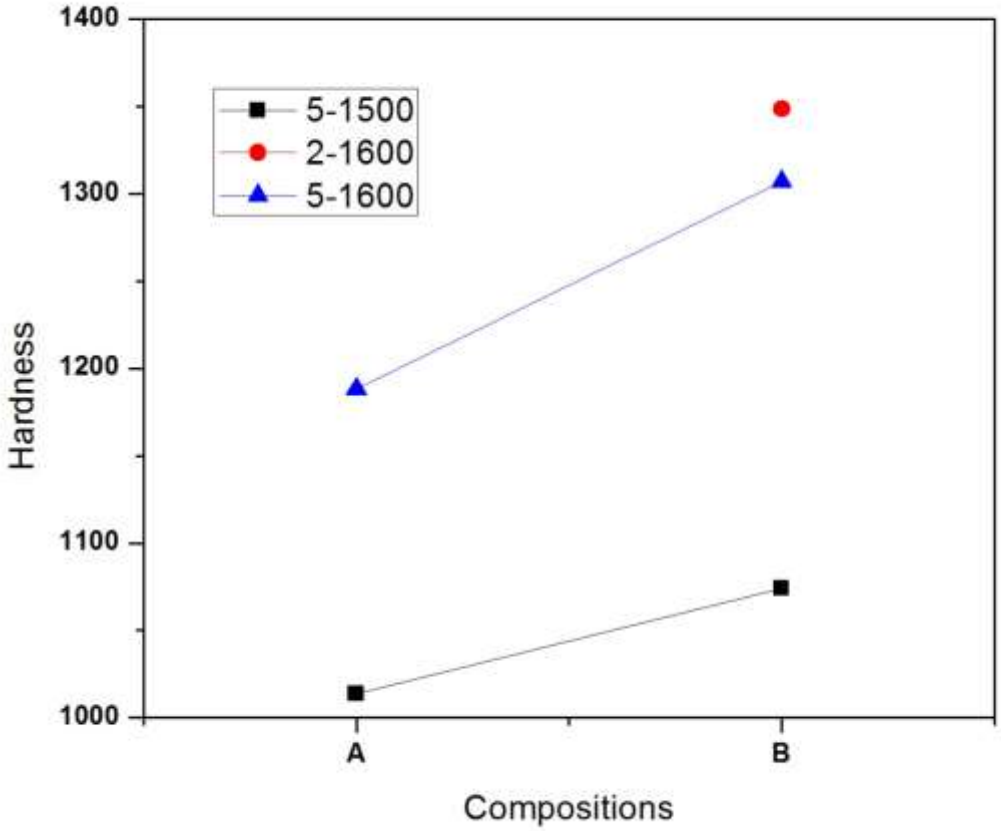
It is observed that with increase in temperature from 1500^{o}C to 1600^{o}C, hardness of batch-4 and batch-5 increased from 1014 kg/mm^2 to 1188 kg/mm^2 and from 1074 kg/mm^2 to 1307 kg/mm^2. This increase in the value of hardness may be attributed to higher density observed at 1600^{o}C than 1500^{o}C. Silica source (MK/Pure Si) didn't affect much the hardness value at 1500^{o}C and 1600^{o}C. However the percentage of silica source (MK/Pure Si) did affect the hardness value. The hardness value increased from 1074 to 1307 kg/mm^2 when volume percentage of silica source (Pure Si) was decreased from 5 to 2.

Hardness value increases with decreasing in amount of silica or polymer content silica and for increasing temperature.

Load=10 kg-f

Holding time=10 sec

SAMPLE NAME	**COMPOSITION**	**TEMPARATURE (^{O}C)**	**HARDNESS(kgf/mm²)**			**Avg value**	**DIAGONAL VALUE(mm)**			**Avg value**
BATCH-4	95-Al_2O_3,5-MK	1500	991.6	1029.7	1020.6	1013.9	136.7	134.19	134.7	135.1
BATCH-5	95-Al_2O_3,5-SI	1500	1072.4	1098.1	1051.2	1073.9	131.49	129.9	132.81	131.36
BATCH-6	98 Al_2O_3,2-MK	1600	1361.8	1302.9	1335.7	1333.4	116.69	119.30	117.82	117.9
BATCH-7	98- Al_2O_3,2-SI	1600	1366.3	1327.3	1352.2	1348.6	116.58	118.19	117.10	117.2
BATCH-4	95 Al_2O_3,5-MK	1600	1156.1	1198.3	1210.1	1188.1	126.64	124.39	123.79	124.8
BATCH-5	95- Al_2O_3,5-SI	1600	1288.9	1300.8	1331.4	1307	119.94	119.39	118.01	119.06



Make three graphs: 1. 5% Si and MK at 1500°C, 2. 2% Si and MK at 1600°C and, 3. 5% Si and MK at 1600 °C.

CHAPTER-5

CONCLUSIONS

- It was observed that as the dwelling time is increased from 1 hr to 10 hr the density also increases and the apparent porosity minimized. Maximum density (3.6964g/cc) and is observed at 1500^{o}C for 6 hr.
- With increase in temperature from 1500^{o}C to 1600^{o}C the density increased from 3.69 g/cc to 3.83 g/cc for Batch-7.
- Porosity also decreased from 39.25 % to 5.65% when dwelling time during sintering was varied from 1 hr to 6 hr at 1500^{o}C
- Porosity also decreased from 13.45% to 3.40 % when dwelling time during sintering was varied from 1 hr to 6 hr at 1600^{o}C
- With increase in MK conc. (from 2 to 5%) SEM images showed less haziness. With increase in amount of silica (MK/Pure Si) the grain size decreased.
- Dilatometric studied showed that maximum shrinkage occurred for batch-2 and minimum shrinkage for batch-3,4 and 5.
- Maximum hardness was observed for batch-7 and minimum for batch-4.

Thus it can be concluded that by changing the silica source (MK/Pure Si), the properties (AP, BD, Density, Shrinkage, Hardness) hardly changed. However, on changing the percentage of MK polymer/Pure Si , the values got changed.

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